## **Report on the experiment HC-3347**

Investigation of Laue transmission case in diamond compound refractive lenses

## Summary

During the beamtime, four different types of diamond plates were tested (fig. 1). They were made from different diamond materials and had different material quality in terms of purity, crystallographic orientation and ideality. Most of them were laser-cut and had compound X-ray refractive lenses (CRLs), fig. 1 (b-d). They were investigated according to the logic explained in the proposal - we used EXAFS mode and a so-called 'glitch effect' under various conditions has been registered. We were varying position and orientation of samples, using different divergence of incident radiation. With the aid of orientation matrix obtained during the current beamtime, we became able to theoretically calculate glitches to minimize them in single-crystal refractive lenses. The last is of high interest for front-end and beam-conditioning applications.



**Figure 1.** Four types of diamond plates used for the experiment – no profiling (a), onedimensional CRLs made from slightly different diamond materials (b-c) and two-dimensional lens (d). Red arrows show crystallographic directions. Dimensions are in mm.

## **Experimental details**

The experiment was done at the BM31 of the ESRF (fig. 2). Before the main experiment, we did a calibration of the MAR345 diffraction detector with lanthanum hexaboride (LaB6 660c). We defined some key parameters like sample-to-detector distance and center positions for both X and Y axis. MAR345 detector has relative large active area with diameter of up to 345 mm and pixel size of 0.1mm. MAR345 detector was later used in the current experiment for reconstruction of the orientation matrix of all samples (fig. 3).





**Then,** we tested two samples from the figure 1 (a, b). These plates were identical and grown by chemical vapor deposition (CVD). They were supplied by Element Six Ltd (Ascot, UK; product reference No. 145-500-0248). Additionally, X-ray refractive lenses were laser-cut in the second diamond plate, as can be seen in Fig. 4 (a).

During diffraction measurements [010] crystallographic direction of both plates was along the X-ray beam (fig.1, b). This direction will be further called as  $\chi$ -axis. We performed EXAFSmeasurement when during the continuous energy scan the X-ray intensity gets detected by ion chambers both before and after the sample (I<sub>0</sub> and I<sub>1</sub>, respectively). The step of the scan was limited by the resolution of the monochromator of 1 eV. Glitches emerging in normalized I<sub>0</sub> spectrum are considered as noise signal and are monochromator glitches due to multiple scattering. Sample-induced glitched remain in I<sub>1</sub>. Dividing I<sub>1</sub> by I<sub>0</sub> one can get rid of the monochromator glitches, leaving only the meaningful signal from the sample. Some other specific steps of data manipulation are listed as follows:

- 1) Plot  $I_1/I_0$  division as a function of the X-ray energy.
- 2) Low-polynomial (1 or 2, max 3) fitting through the remaining  $I_1/I_0$  curve. This fit would mark the reference line.

3) The glitch dip is then given in percents with respect to the baseline.





**Fig. 3.** Images collected from MAR detector will allow to reconstruct the orientation matrix for all used samples and mathematically calculate glitches. Data treatment is in progress.



Fig. 4. Optical micrograph of two diamond plates used for experiment and described in this report.

The X-ray absorption spectra obtained from the first diamond plate (fig 1, a) was used as a reference one as no CRLs are cut in this plate. The interest is to later compare spectra from all of the samples (fig. 1). Nevertheless, for the first sample we launched two scans: one - under the configuration of  $\chi=0^{\circ}$  and then another one - by rotating 30° clockwise around  $\chi$ -axis. Fig. 5 shows that two curves are almost similar except a slight shift in energy positions of glitches. In addition, glitches appearing for the case of  $\chi=30^{\circ}$  have less amplitude compared with those of  $\chi=0^{\circ}$ . The calculation of this effect is in progress.



Fig. 5. Two EXAFS spectra of the diamond plate with two different  $\chi$  angles.

The second plate (fig. 1, b) has two sets of compound refractive lenses, they are denoted as  $CRL_5$  and  $CRL_2$  respectively (fig. 6, a). The subscript indicates number of biconcave lenses. The term 'bulky material' refers to the part with no profiling between  $CRL_5$  and  $CRL_2$ . EXAFS measurements were done consistently for the center of  $CRL_5$  and for the bulky part.



Fig. 6. a) Schematic drawing of diamond lenses with two arrays of compound lenses and buly material in between. b) The condenser CRL.

To increase signal-to-noise ratio, we repeated each EXAFS scan three times in the energy range from 16 keV to 18 keV. These curves were then averaged and are shown in fig. 7 (a,b). It can be seen that glitches occurring in Fig. 7 (a) are not so pronounced as those in Fig. 7 (b). This phenomenon could be attributed to the fact that X-ray beam traverses larger distance in the former case compared with the latter one and more intensity is attenuated due to absorption.



**Fig. 7.** Plot of intensity vs energy: X-ray impinges on the center of CRL<sub>5</sub> without condenser (a) and with (c). X-ray impinges on the bulky part without condenser (b) and with (d).

Additionally, we check if beam divergence plays a significant role for diffraction conditions (that in turn affects the transmitted spectrum). For this purpose, we installed a condenser (fig. 6, b) consisting of five 2D beryllium lenses with the radius of curvature of 50  $\mu$ m. Focal length of the condenser was f = 1.5 m at E = 10 keV and f = 6.5m at the E=20 keV, giving the divergence of 170 urad and 40 urad, respectively. Fig. 7 shows that averaged glitch scans are identical for both cases: with and without condenser (a-c and b-d). In other words, beam divergence is a non-functional factor.

Different scanning positions might also influence the transmission spectrums significantly, and in particular, the amplitude of glitches. To check this idea, we made three different scans when thin 200-um size X-ray beam (in vertical direction) was interacting with the center of the  $CRL_5$ , its edge and bulky material, respectively. Fig. 8 indicates that energy positions for three glitch scans are identical except the amplitude of the glitches – central part has smallest drops. This can be explained by absorption as thickness of the material is getting larger while shifting to the edge of the lens and has its maximum at the bulky part.



Fig. 8 Various location of the incident beam with respect to CRL<sub>5</sub> affects EXAFS spectrums.

## Outline

We also did some more measurements with other samples (fig. 8). A particular interest to us is 2D diamond lenses as they have high potential for front-end applications. By the moment these results as well as theoretical calculations are still in progress and can not be presented in the current report. We plan to complete and publish all the results of the experiment in 2018.